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L9 and L2	5

Database: US Pre-Grant Publication Full-Text Database; US Patents Full-Text Database; US OCR Full-Text Database; EPO Abstracts Database; JPO Abstracts Database; Derwent World Patents Index; IBM Technical Disclosure Bulletins

Search: L10

Refine Search | Recall Text | Clear | Interrupt

Search History

DATE: Thursday, September 02, 2004 Printable Copy Create Case

Set Name side by side	Query	Hit Count	Set Name result set
DB=PGPB,USPT,USOC,EPAB,JPAB,DWPI,TDBD; PLUR=YES; OP=ADJ			
L10	L9 and l2	5	L10
L9	L8 with (water or H$1O)	622	L9
L8	L5 with ag with v with o	8619	L8
L7	L5 same ag same v same o	81481	L7
L6	L5 and ag and v and o	220837	L6
L5	(li or an or k or rb or cs or ti or mg or ca or sr or ba or cu or zn or cd or pb or cr or au or al or fe or co or ni or mo)	23635849	L5
L4	L3 and ag and v and o	540	L4
L3	l1 and (li or an or k or rb or cs or ti or mg or ca or sr or ba or cu or zn or cd or pb or cr or au or al or fe or co or ni or mo)	3306	L3
L2	L1 and (502/$ or 429/$ or 423/$ or 502/$ or 549/$)	1522	L2
L1	silver and vanadium and metal oxide and catalyst	3312	L1

Hit List

Clear | Generate Collection | Print | Fwd Refs | Bkwd Refs | Generate OACS

Search Results - Record(s) 1 through 5 of 5 returned.

☐ 1. Document ID: US 20040116737 A1

Using default format because multiple data bases are involved.

L10: Entry 1 of 5 File: PGPB Jun 17, 2004

PGPUB-DOCUMENT-NUMBER: 20040116737
PGPUB-FILING-TYPE: new
DOCUMENT-IDENTIFIER: US 20040116737 A1

TITLE: Nox treated mixed metal oxide catalyst

PUBLICATION-DATE: June 17, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Gaffney, Anne Mae	West Chester	PA	US	
Heffner, Michele Doreen	Chalfont	PA	US	
Song, Ruozhi	Wilmington	DE	US	

US-CL-CURRENT: 562/546; 502/302, 562/547

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw De

☐ 2. Document ID: US 5881358 A

L10: Entry 2 of 5 File: USPT Mar 9, 1999

US-PAT-NO: 5881358
DOCUMENT-IDENTIFIER: US 5881358 A

TITLE: Composition for extracting transition metal and method for extracting transition metal using the same

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw De

☐ 3. Document ID: US 4272409 A

L10: Entry 3 of 5 File: USPT Jun 9, 1981

US-PAT-NO: 4272409
DOCUMENT-IDENTIFIER: US 4272409 A
** See image for Certificate of Correction **

TITLE: Aluminosilicate sols and powders

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw De

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☐ 4. Document ID: US 4257874 A

L10: Entry 4 of 5 File: USPT Mar 24, 1981

US-PAT-NO: 4257874
DOCUMENT-IDENTIFIER: US 4257874 A
**** See image for Certificate of Correction ****

TITLE: Petroleum refinery processes using catalyst of aluminosilicate sols and powders

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw De

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☐ 5. Document ID: US 4217240 A

L10: Entry 5 of 5 File: USPT Aug 12, 1980

US-PAT-NO: 4217240
DOCUMENT-IDENTIFIER: US 4217240 A

TITLE: Stable aluminosilicate aquasols having uniform size particles and their preparation

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw De

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Clear | Generate Collection | Print | Fwd Refs | Bkwd Refs | Generate OACS

Terms	Documents
L9 and L2	5

Display Format: - Change Format

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d his

     (FILE 'HOME' ENTERED AT 17:26:00 ON 02 SEP 2004)

     FILE 'CAPLUS' ENTERED AT 17:28:02 ON 02 SEP 2004
L1            55 S MULTIMETAL OXIDE
L2            39 S MULTI-METAL OXIDE
L3            92 S L1 OR L2
L4         60239 S METAL OXIDE
L5         60280 S L4 OR L3
L6            83 S L5 AND AG AND V AND O
L7       6200095 S LI OR MA OR K OR RB OR CS OR TL OR MG OR CA OR SR OR BA OR CU
L8            75 S L7 AND L6
L9            14 S L8 AND (WATER OR H2O)
L10            9 S L9 AND PY<1998

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L10 ANSWER 1 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1997:381724 CAPLUS
DOCUMENT NUMBER: 127:141945
TITLE: Electrochemistry of the copper-nickel series of heteropolymetallic complexes [(μ4- O){NC5H4[C(O)NEt2]-3}4Cu4-x{Ni(H2O)}xCl6] (x = 0-4)
AUTHOR(S): Workie, Bizuneh; Dube, Christopher E.; Aksu, Levent; Kounaves, Samuel P.; Robbat, Albert, Jr.; Davies, Geoffrey
CORPORATE SOURCE: Dep. of Chem., Tufts Univ., Medford, MA, 02155, USA
SOURCE: Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1997), (10), 1739-1745
CODEN: JCDTBI; ISSN: 0300-9246
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
AB The electrochem. of the tetranuclear copper-nickel heteropolymetallic complexes [(μ4- O)L4Cu4-x{Ni(H2O)}xCl6] [x = 0-4, L = N,N-diethylnicotinamide (denc)] were studied at a platinum electrode in DMSO with 0.20M tetrabutylammonium hexafluorophosphate as supporting electrolyte. At potentials more cathodic than -1.0 V the complexes are electrodeposited as Cu-Ni alloy and metal oxide films and display a complicated set of cyclic voltammographs. The voltammographs of all the Cu-containing complexes show a quasi-reversible redox couple in the potential range 0.250 to -0.450 V vs. Ag-AgPF6 (0.01M)-CH3CN. As the number of Cu atoms decreases in the complex, the peak currents ipa and ipc decrease proportionally and the peak potential shifts anodically. The cyclovoltammetric (CV) results indicate that electron transfer initially occurs only to the CuII centers and that the electron-transfer reaction appears to be quasi-reversible. Using steady-state voltammetry at an ultramicroelectrode in combination with chronoamperometry at a microelectrode and exhaustive electrolysis at a Hg-pool electrode, the number of electrons (n) transferred for this initial reduction of the Cu4, Cu3Ni, Cu2Ni2 and CuNi3 complexes were 3.1, 2.1, 1.8 and 0.57, resp. The diffusion coefficient for all the complexes was 2.2(±0.1) + 10-6 cm2 s-1. The electronic spectrum of the Cu4 complex taken after exhaustive electrolysis shows that one quarter of the Cu atoms remain in the CuII form and that the CuI complex remains stable. Since only a single CV peak results for all of the complexes, the electron transfer is most likely consecutive with very closely spaced E° potentials. A model based on statistically determined electron transfer to CuII in particular faces is also proposed.
REFERENCE COUNT: 31 THERE ARE 31 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> d 2-9 ibib abs hitstr

L10 ANSWER 2 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1993:580403 CAPLUS
DOCUMENT NUMBER: 119:180403
TITLE: Preparation of α,β-unsatd. nitriles by catalytic vapor-phase ammoxidation of saturated hydrocarbons
INVENTOR(S): Blanchard, Gilbert; Bordes, Elisabeth; Ferre, Gilbert
PATENT ASSIGNEE(S): Rhone-Poulenc Chimie SA, Fr.
SOURCE: Eur. Pat. Appl., 16 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

EP 547981	A1	19930623	EP 1992-420446	19921203 <--
EP 547981	B1	19950809		
R: BE, DE, ES, FR, GB, IT, NL, PT				
FR 2684989	A1	19930618	FR 1991-15843	19911216 <--
FR 2684989	B1	19940204		
ES 2076727	T3	19951101	ES 1992-420446	19921203 <--
JP 06135921	A2	19940517	JP 1992-351423	19921208 <--
JP 06092355	B4	19941116		
CA 2085451	AA	19930617	CA 1992-2085451	19921215 <--
CA 2085451	C	19970121		
CN 1075314	A	19930818	CN 1992-114404	19921215 <--
CN 1032647	B	19960828		
RU 2060247	C1	19960520	RU 1992-4546	19921215 <--
US 5334743	A	19940802	US 1992-991170	19921216 <--

PRIORITY APPLN. INFO.: FR 1991-15843 19911216
OTHER SOURCE(S): CASREACT 119:180403

AB Alkanes are converted to α,β-unsatd. nitriles by ammoxidn.. in the vapor phase in the presence of solid catalysts, the active phase of which consists of **Mo**, **V**, and **O**, along with at least one of the elements Mn, **Zn**, **Co**, **Cu**, **Li**, Na, **K**, or **Ag**. The process is particularly useful for the ammoxidn.. of propane to give a mixture of acrylonitrile and propylene. Thus, catalysts such as Mn0.8V1.6Mo0.4O6, Zn0.85V1.7Mo0.3O6, or Co0.9V1.7Mo0.2O6 (prepns. given) were used in the ammoxidn.. of propane (NH3, O2, **H2O** vapor, He flow, 1.3 bar) at various temps. to afford acrylonitrile and propylene mixts.

L10 ANSWER 3 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1991:186328 CAPLUS
DOCUMENT NUMBER: 114:186328
TITLE: Molybdenum- and phosphorus-based oxidation catalysts for the production of methacrylic acid from methacrolein, isobutyraldehyde, and isobutyric acid
INVENTOR(S): Kurimoto, Ikuo; Hashiba, Hideto; Onodera, Hideo; Aoki, Yukio
PATENT ASSIGNEE(S): Nippon Shokubai Kagaku Kogyo Co., Ltd., Japan
SOURCE: Eur. Pat. Appl., 32 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 415347	A2	19910306	EP 1990-116480	19900828 <--
EP 415347	A3	19920226		
EP 415347	B1	19941026		
EP 415347	B2	20020605		
R: BE, DE, ES, FR, GB, IT, NL				
JP 03086242	A2	19910411	JP 1989-220380	19890829 <--
JP 04090853	A2	19920324	JP 1990-206857	19900806 <--
JP 2994706	B2	19991227		
US 5153162	A	19921006	US 1990-574139	19900829 <--

PRIORITY APPLN. INFO.: JP 1989-220380 A 19890829
JP 1990-206857 A 19900806

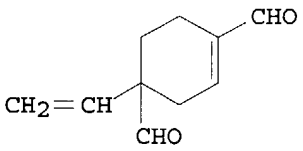
AB Catalysts for the title use contain an active layer of MoaPbAcCeBdDfOx (I, A = As, Sb, Ge, Bi, Zr, Ce, or Se; B = **Cu**, **Fe**, **Cr**, **Ni**, Mn, **Co**, Sn, **Ag**, **Zn**, Pd, Rh, or Te; C = **V**, W, or Nb; D = alkali metal, alkaline-earth metal, or **Tl**; a, b, c, d, e, f, and x = atomic ratio for each atom; when a = 12, b = 0.5-4, c = 0-5, d = 0-3, e = 0-4, f = 0.01-4, x = number of oxidation states of all elements) and are prepared by mixing compds. of the elemental components of the oxide of I, heating the mixed compds. to prepare a slurry or a solution, spraying the slurry or solution onto an inert carrier, and calcining. Adding arsenious acid 123.7, 85% H3PO4 523.8, and CsNO3 812.3 g in 4 L HNO3 and 5 L **H2O** to 8830 g NH4 paramolybdate 8830 and 531.4 g NH4 metavanadate, stirring, spraying the heated slurry onto a

silicon carbide carrier, and calcining at 400° for 3 h gave a catalyst with composition Mo12P1.09V1.09Ce1.0As0.3Ox. A vapor mixture containing methacrolein 3.5, isobutylene 0.04, methacrylic acid (II) and acetic acid 0.24, steam 20, O 9.0, and others 67.2 volume% was oxidized with this catalyst to give II with 87.1% selectivity and 89.2% conversion.

L10 ANSWER 4 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1985:166979 CAPLUS
DOCUMENT NUMBER: 102:166979
TITLE: Vinylcyclohexenedicarboxaldehydes
PATENT ASSIGNEE(S): Nippon Zeon Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 59204148	A2	19841119	JP 1983-75282	19830428 <--

PRIORITY APPLN. INFO.: JP 1983-75282 19830428
GI



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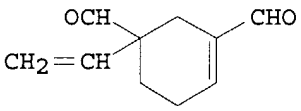

II

AB The title compds. (I and II) were prepared by oxidation of C5-compds. containing Me groups by mol. O in the presence of mixed oxides of (1) Sn, (2) Co, Ni, Se, Bi, P, U, Zr, Cu, Pb, Mn, W, V, and/or Cr, (3) groups I and II and/or Tl, (4) Al, In, Mb, Ta, Ga, Th, Pd, Ru, Rh, Ge, Ag, B, As, Se, Si, Ti, Mo, Te, Sb, and/or rare earth metals. Thus, a small quantity of concentrated HNO3 was added to a mixture of 118.7 g Sn and 100 mL H2O, the resulting mixture stirred, evaporated to dryness, dried at 120° for 8 h, calcined at 350° for 4 h, pulverized to < 100 mesh, 11.5 g 85% H3PO4 and 200 mL H2O were added, the resulting mixture was dried at 120° for 8 h, calcined at 350° for 4 h, pulverized to < 100 mesh, adsorbed to about 30 wt % on a silicon carbide support, heated at 400° for 2 h, and then calcined at 900° for 6 h to give a Sn1P0.1 catalyst, over which (50 mL) in a reactor heated at 470° was passed (SV 3500 h-1) a 2:20:78 (molar ratio) mixture of tert-amyl alc., air, and steam to give a mixture containing 5.7% I and 94.3% II with 31% selectivity and 42% conversion.

L10 ANSWER 5 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1984:571916 CAPLUS
DOCUMENT NUMBER: 101:171916
TITLE: Unsaturated acids and esters by oxidative condensation
INVENTOR(S): Grasselli, Robert Karl; Guttmann, Andrew Tytus
PATENT ASSIGNEE(S): Standard Oil Co., USA
SOURCE: Eur. Pat. Appl., 20 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 111605	A1	19840627	EP 1982-306820	19821221 <--
EP 111605	B1	19860409		

R: BE, CH, DE, FR, GB, IT, LI, LU, NL, SE

CA 1193613 A1 19850917 CA 1982-417257 19821208 <--
PRIORITY APPLN. INFO.: EP 1982-306820 19821221
AB Oxidation catalysts for the selective oxidation of MeOAc [79-20-9] or Me propionate [554-12-1] with MeOH [67-56-1] (or Me2O [115-10-6]) at 330° in the presence **O** (in N dilution) have the general formula AaBbCcPOx, A is alkali metal, alkaline earth metal, or **Tl**; a = 0-1.5; B is **Fe**, **Co**, **Ni**, **V**, **Cr**, Mn, **Cu**, Pt, Pd, Rh, Ru, or **Ag**; (b = 0.01-3.0); C is Sc, Y, Ti, W, Nb, Si, La, Ge, Ce, Th, U, **Mo**, Bi, Sb, or Te; (c = 0-2.0); and x is the number required to satisfy valence requirements. Thus, 44.1 g Al2O3.xH2O in 170 mL **water** at 70° was stirred with aqueous (NH4)2HPO4, aqueous Th(NO3)4.4H2O and **Cu**(NO3)2.3H2O, and aqueous KNO3, the slurry was evaporated at 90° and dried at 120-125°, and the dried material was heated 3 h at 290° and 3 h at 350°, ground, screened to 20-35 mesh, and calcined 5 h at 500°. A fixed-bed reactor containing the catalyst was fed at MeOAc-MeOH-**O**-N ratio 10:1:0.8:7.2 to give 74.9% Me acrylate [96-33-3] and 10.9% acrylic acid [79-10-7].

L10 ANSWER 6 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1971:124849 CAPLUS
DOCUMENT NUMBER: 74:124849
TITLE: Acetic acid
INVENTOR(S): Nakajima, Kazuhisa; Sato, Tsuneo
PATENT ASSIGNEE(S): Japan Synthetic Chemical Industry Co., Ltd.
SOURCE: Jpn. Tokkyo Koho, 3 pp.
CODEN: JAXXAD
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 45041571	B4	19701226	JP	19671213 <--

AB Butene is oxidized at 220-350° with **O**-containing gases in the presence of steam over VXO catalysts (X = **Cr**, Mn, **Cu**, **Zn**, Bi, **Ag**, **Pb**, Zr, and (or) Nb and **V** -X = 1:0.01-1.5). Thus NH4VO3 and **Cr**(AcO)3.**H2O** in hot **H2O** is mixed with silica colloid aqueous solution, evaporated, heated to 480° during 8 hr in air, roasted 8 hr at 480°, and crushed to give a catalyst with **V**-**Cr**-**O** 1:0.1:2.8. Oxidation of butene-air-steam mixture over this gave 49.2% AcOH.

L10 ANSWER 7 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1970:54769 CAPLUS
DOCUMENT NUMBER: 72:54769
TITLE: Catalysts for production of acrylic acid by vapor-phase oxidation of acrolein
PATENT ASSIGNEE(S): Toa Gosei Chemical Industry Co., Ltd.; Institute of Physical and Chemical Research
SOURCE: Brit., 8 pp.
CODEN: BRXXAA
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1170851		19691119		<--
DE 1643087			DE	
US 3567772		19710000	US	<--
PRIORITY APPLN. INFO.:			JP	19660330

AB Catalyst complexes consisting of mixts. of **Mo**, **V**, and **Al** oxides and oxides of **Cu**, Ge, U, Mn, **Ba**, Si, **Au**, Se, **Ca**, Ti, **Sr**, **Ag**, **Mg** , B, Sn, W, **Pb**, or As are used for the vapor-phase oxidation of acrolein (I) to give high yields of high-purity acrylic acid (II). Thus,

92.7 g 6-10 mesh **Al** sponge was warmed with an aqueous solution containing NH4 molybdate 22.45, NH4VO3 2.36, and **Cu**(NO3)2 5.52 g and the mixture was stirred and dried at 120° before being heated in air at a temperature increasing to 285°. The catalyst was screened and 50 ml was replaced in the reaction tube and heated to 300°. A feed stream containing 3.2% I, **O** at a mole ratio of 2.4, and **H2O** at a **mo** le ratio of 15 with respect to I, was introduced at 1230 hr-1 space veloc ity and the temperature was increased to 400°. The oxidation was continued for 3 hr over a 100:10:3:10 MoO3-V2O5-Al2O3-CuO catalyst and a 99.9% conversion was obtained giving a 91.1% yield of 95.2% pure II. Conversions of 99.8 and 98.4% were obtained at 325 and 300°, resp., giving yields of 95.7 and 96% of 97.3 and 97.6% pure II. Other catalysts used contained GeO2, UO3, MnO2, BaO, SiO2, Au2O3. SeO2, CoO, SrO, CuO mixed with GeO2, Ag2O, MgO, SnO, TiO2, B2O3, WO3, As2O5, and PbO in place of CuO.

L10 ANSWER 8 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1968:49085 CAPLUS
DOCUMENT NUMBER: 68:49085
TITLE: Citric acid
INVENTOR(S): Wiley, Richard H.
SOURCE: U.S., 4 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3356721		19671205	US	19620321 <--

GI For diagram(s), see printed CA Issue.

AB The preparation of the title compound (I) comprised (a) air oxidation of a 2-butene-1,4-dioic acid over a Pd salt catalyst to give the corresponding oxalacetic acid di-(lower alkyl) ester or anhydride; (b) condensing the ester or anhydride with **O**:C:CH2 (II) in the presence of a ZnCl2-HOAc complex, BF3, or Al2O3-SiO2-B2O3 catalyst to form the β-lactone; (c) hydrolysis of the β-lactone with **Ca** (OH)2 to obtain the **Ca** salt, and subsequent treatment with H2SO4. Di-Me fumarate 10 was blown with air 1-5 hrs. in the presence of CuCl2-Pd catalyst 0.1 part, until a maximum concentration of the di-Me oxalacetate, MeCO2CH2COCO2Me, (III) was obtained. III was identified by spectrometric anal. and the 2,4-dinitrophenylhydrazone, m. 110° (decomposition). The catalyst was separated by filtration and ion exchange treatment, the resulting mixture (containing 40-95% III) treated with 0.5 part ZnCl2-HOAc complex and II (obtained from diketene) in an amount equivalent to the III present, kept 1 hr. to complete reaction, and the resulting intermediate (β-lactone), 4-carbomethoxy-4-carbomethoxymethyl-2-oxetanone (IV) hydrolyzed with **Ca**(OH)2 to give **Ca** citrate, which on acidification with H2SO4 yielded I. IV might be isolated prior to hydrolysis by distillation in vacuo; IV heated to 40-60° gave a polymeric product, which with heat yielded the di-Me aconitate and, and itaconate resp. An Et2O solution of the Et analog of III (**V**) containing 16 g. ester was obtained by the Et2O extraction of an aqueous solution of Na oxalacetate, neutralized with dilute H2SO4, and the mixture treated with BF3-etherate (0.2-0.5 weight % fluoride), then with 4.2 g. II at 0-10°, the mixture kept an hr., and the Et2O evaporated at low temperature to give the Et analog (IV) of IV. VI on controlled reaction with an equivalent amount cold **H2O** was converted to asym. di-Et citrate (VII) (**Ag** salt m. 110°) which was isolated as a syrup. VII was converted to its Ac derivative, to citric acid, or to tri-Me citrate. Alcoholysis (EtOH) of VII gave tri-Et **O** -ethylcitrate. VI heated lost CO2 to give di-Et itaconate on fractionation. Heating the crude oxetanone with a trace of Bz2O2 initiator gave a foamed polymer of di-Et itaconate. Hydroxymaleic anhydride (VIII) analogously treated gave the β-lactone intermediate, 1,6-dioxaspiro[3.4]octane-2,5,7-trione (IX). IX warmed gently to .apprx.25° with **H2O** yielded I. VIII, m. 81-2° 11 (prepared by reaction of diacetyltartartic anhydride with C5H5N at -5° was dissolved in Et2O 80 parts treated with an equivalent amount of

II in the presence of BF3 (1.0 weight %) at 0-5° gave IX, which was recrystd. at low temperature A stirred solution of 17 g. **V** in 10 ml. Et2O over 0.5 g. Al2O3-SiO2-B2O3 catalyst (U.S. 2,462,357) was treated with 4.2 g. II at 20-5° to give pure IV, as a semisolid which hydrolyzed to I. IX was similarly obtained to give I, m. 98-100°. Air was passed through a suspension of 0.2 g. fumaric acid in 5 ml. concentrated HCl containing 0.1 g. PdCl2, during 15 min. An aliquot portion of the solution gave the 2,4-dinitrophenyl-hydrozone of oxalacetic acid, m. 170°. Decationization of the reaction mixture on an ion exchange resin followed by evaporation gave pure oxalacetic acid, m. 150.2° (EtOAc). A solution of 10 g. maleic anhydride in 1 l. C6H6 was allowed to flow downward over 200 ml. supported Pd catalyst in the presence of a rising stream of air during 4 hrs. The catalyst was prepared by converting a cation exchange resin (Dowex 50-X8, a sulfonated styrene) to the Pd salt by exchange with a PdCl2 solution or depositing PdCl2 or PdSO4 over a **metal oxide** on alumina-silica, alumina-zirconia, or alumina-titania. The effluent C6H6 was treated with 0.5 ml. BF3 etherate and 4.2 g. II to give IX. The depalladinized solution was distilled in vacuo to sep. the solvent, the reactant, and **V**.

L10 ANSWER 9 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1910:5058 CAPLUS
DOCUMENT NUMBER: 4:5058
ORIGINAL REFERENCE NO.: 4:867i,868a-i,869a
TITLE: Latest Methods of Manufacturing Metallic Lamp Filaments
AUTHOR(S): Duschnitz, B.
SOURCE: Elec. Rev. and West. Elect. (**1910**), 56, 32-33,89-91
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB An abstract and r.acte.esum.acte.e of foreign patents on the subject during the past year. Processes are divided into three divisions: (1) "With the aid of organic substances." (2) "From substances free from C by the wet process." (3) "From metallic masses free from C without the use of fluids." (2) is subdivided into (a) "with colloids" and (b) "from combinations of fluid substances." (1) The Wolfram Lamp **Co**., of Augsburg, Germany, patent a filament of a mixture of W and a small quantity of more easily fusible metals for sintering it, as **Cr**, **V**, Nb, Ta, Os, Ru, Zr, or Th. They claim greater strength and elasticity. W powder with 15% or more of above metals is mixed with a ZnCl2 or acetic acid solution of cellulose, formed into filaments, carbonized and the C removed by one of the regular processes. Siemens and Halske patent the removal of C from filament by heating in an atmos. of acetic acid at reduced pressure. The decarbonization is said to be very rapid and thorough. E. Goosens Pope & **Co**., Holland, mix a Na tungstate solution with not more than 8% gallic acid and precipitate with acid. The precipitate is a mixture of low W oxides and pyrogallol. It is washed with **water** and 96% alc. The black shiny paste obtained is formed into filaments, dried slowly, heated to 600° in CO2 leaving the filament a mixture of W **metal**, **oxide** and carbide, then in H2 to reduce oxide and decompose carbide. The French Auer **Co**., winds the filament pressed from a mixture of W and organic binder on the mount, fires in special furnace in H2 and completes sintering by passing current through the filament. The mount is then placed in N and filaments glowed to drive H from metallic parts of mount. If this is not done the occluded H escapes slowly during burning and gives a bad vacuum. The Zirkon Lamp **Co**. remove carbon by means of N or nitrogenous gases produced in vacuum from H-and N-containing P comps., as phospham, phosphamides and phosphamine or their sulphates. Phospham (PN2H) is especially suitable, decomposing at low temperature and liberating N and P. The process is carried out by (1) mixing the phospham with the metal and binder before pressing, or (2) dipping the filaments in an alc. suspension of phospham before sealing in or (3) dusting the filaments with phospham. Decarbonization may be done during exhaustion. (2a) Johann Lux, of Vienna, presses a paste made of colloidal tungstic acid and W powder. After drying, current is passed through the filaments in an atmos. of H to reduce acid to W. Hans Kuzel finds that the addition to his regular colloid of 5-20% of oxide or hydroxides of refractory metals increases the cohesion of the wet

as well as the dry material. Hollow spaces are quite frequent in the filaments. To avoid this he glows them in an inert or reducing atmosphere at a pressure of not more than 150 mm. Hg. The Siemens and Halske Co., find that plastic acid NH4 tungstate becomes conducting and insol. in H2O if heated below red heat in an atms. free from O. They now prepare the plastic mass by evaporating NH4 tungstate solution, acid NH4 tungstate separating out. This is filtered off and heated gradually to 120-150°, then to 270° in vacuum (20 mm.Hg). Resulting powder is put in hot H2O and evaporated to dryness. Residue is a glassy mass. This can be dissolved in hot H2O and pressed into filaments. J. Lux, Vienna, prepares a binder from NH4 metatungstate, mixes it with W metal and glows in a reducing atmosphere. (3) Siemens and Halske patent the drawing of Zr and Th or mixtures of these with other metals in a ductile tube of another metal, removing the envelope after drawing by chemical or mechanical means or fusion. They use a spongy mass of Th, etc., obtained by making alloys, and dissolving out the alloying metal. Filaments from a spongy mass are said to be more easily workable than those from powder. The same firm patents the use of powdered W in connection with low evaporating metals as Ag, Ni, Au nd Pt. These metals coat the W and the coated W may be pressed into rods and drawn into wire. The low evaporating metal is removed by passing a current. The W powder may be pressed into a tube of Ag, Ni, etc., and the whole encased in an outer tube of steel. This is drawn, the outer tube removed by solvents, the filaments shaped and the low evaporating metal removed by passing a current through the filaments. They also patent a method of making small filaments by heating large filaments in a mixture of P vapor and H at reduced pressure, the cross section being uniformly reduced. The All. Eleckt. Ges. mix W with low m. p. metal as Cu, Cd, Bi, etc., press in a mold, draw, and remove low m. p. metals by a passing current.